November 1991 A Cahners Publication

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Research & Development®

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R&D

The International Magazine of Research and Development

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ABOUT THE COVER Forensic laboratories are becoming increasingly involved in drug testing. For more on this, see article on page 48.

R&D FOCUS

GOTCHA!!! Forensic Labs Get Smarter 48

Analytical instruments make it easier to identify the smallest thread of criminal evidence.

Self-Regulating Drug Delivery Makes Treatment Automatic 64

Controlled delivery systems are creating new approaches to patient therapy by integrating diagnostics, drugs, and devices.

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Using WorkBench, researchers were readily able to develop a versatile capillary HPLC system.

R&D BUSINESS

Survey Reveals Monthly Changes in R&D

The confidence level that corporate executives have in their future R&D spending, employment stability, and capital spending is reflected in a new business index from Cahners Economics (CE), Newton, MA.

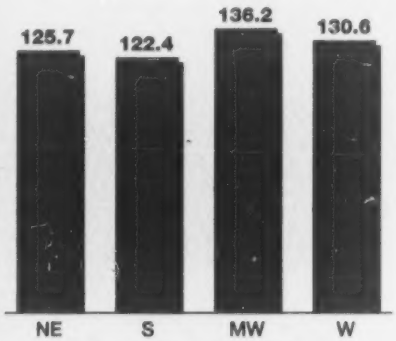
The index is based on 400 monthly interviews with marketing and management people in manufacturing, consumer products, construction, electronics, and computer sectors.

Respondents evaluate the economic environment of their businesses for the next three months and report plans for changes in seven areas: capital equipment purchases, employment, inventories, marketing, new product introductions, and R&D.

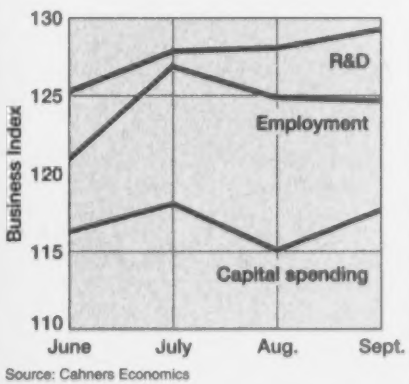
Results from the first surveys reveal that spending for R&D and new product introductions increased steadily from June to September. Spending for capital equipment, marketing, and production was erratic during this period, but the average of all seven indexes in September was about 4% higher than that in June.

Subindex values for regional, company size, and industry groupings also are collected. Regional and industrial grouping indices reveal that corporate managers are confident that R&D spending will continue or improve in all areas of the country and in all industrial sectors. —*Tim Studt*

Improving Economic Conditions Predicted



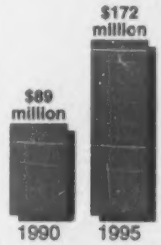
U.S. Regional R&D Indicator
September 1991



Source: Cahners Economics

***Corporate executives** are confident that most aspects of their businesses are positive (indexes >100), but will spend more on R&D than equipment.*

Strong Growth in Fluorescence



With 14.1% annual growth through 1995, fluorescence spectrophotometers are forecast to be the fastest growing market of all the molecular spectroscopy instruments, according to Strategic Directions Intl., a Los Angeles-based management consulting firm.

Biological research is the most important application of fluorescence spectrophotometers, particularly for research-grade instruments.

Biotech Needs New Approval Approach

At least 132 biotechnology-based drugs currently are being developed by U.S. companies, a 63% increase over those being developed in 1988, according to a recent study by the Pharmaceutical Manufacturers Assn. (PMA), Washington, DC.

"Unfortunately, only three biotech drugs were approved by the U.S. Food and Drug Administration over the past 16 months and only 14 drugs were approved over the past nine years," says Gerald Mossinghoff, president of the PMA. "This rate must be increased to keep up with our industry's growing research activities."

A whole new approach to FDA approval must be established to account for the multiple indications produced by biotech-based drugs, according to James Grant, CEO of T-Cell Sciences, Cambridge, MA.

Biotechnology Medicines in Development, is available from Communications Div., PMA, 1100 15th St., NW, Washington, DC 20005. □

New Programs Begin For EC's Framework

The third generation of the European Community's (EC's) R&D Framework Program runs through 1994 and includes several new research programs.

In addition to established programs in information technologies and energy, new programs in the environment, telecommunications, and biotechnology are being established.

The Framework Program has a $2.3 billion budget, which becomes available during late 1991 or early 1992. About $150 million is allocated for telecommunications, $165 million for environmental research, and $65 million for biotech research.

The environmental program includes research studies in global change, environmental monitoring, economic and social aspects of environmental issues, and technological risks of environmental policies.

The biotech program will cover the period 1992 to 1996. It will complement an existing program that ends in 1993, but will have more emphasis on basic biology. The program is open to all organizations in EC member states. The European Commission provides for 50% of the total cost of for shared-cost projects and 100% of the marginal costs for universities.

Additional information from the Commission of the European Communities, Brussels, 010/322-235-1111. □

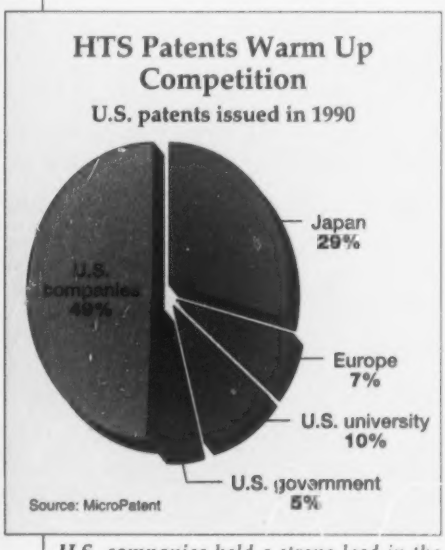
Business continues

Superconducting Materials Find Product Niches

Processing problems have slowed development of high-T_C materials, but application-specific products are starting to emerge.

*By **Tim Studt**, Senior Editor, R&D Magazine*

R&D



***U.S. companies** hold a strong lead in the patents issued for high-T_C materials, apparatus, and processes.*

They may not be fully understood. They may involve complex processing and take several days to heat treat properly. But high-T_C superconducting materials, in both bulk and thin-film forms, are starting to be developed into real-world products.

Some product applications, such as liquid level sensors for cryogenic nitrogen or magnetic bearings, are not readily identified with the zero resistivity inherent in superconductors. Many other applications, though, are directly relatable, such as coils and electric motor windings, infrared sensors, and interconnecting leads to existing low-T_C MRI (magnetic resonance imaging) coils.

The three primary high-T_C materials that researchers are working with are Y-Ba-Cu-O (95 K T_C), Bi-Pb-Sr-Ca-Cu-O (108 K), and Tl-Ca-Ba-Cu-O (125 K). A vanadium oxide-based material with a 130 K T_C has been developed at Hitachi's Superconductivity Research Center, Tokyo, but it is unstable and loses its superconductivity after a few days when exposed to air.

Development of these materials into thin films for sensors and electronic applications, while slow, has been relatively steady, without the major engineering problems that the bulk materials have had to overcome. Development of the bulk materials is considered essential for large-scale commercial and industrial uses.

Bulk high-T_C problems involve the inherent brittleness of the ceramic components, which makes them hard to form into wire. They also have a limited current carrying capacity, and they exhibit "flux creep," which expends electrical energy by opposing lines of magnetic force.

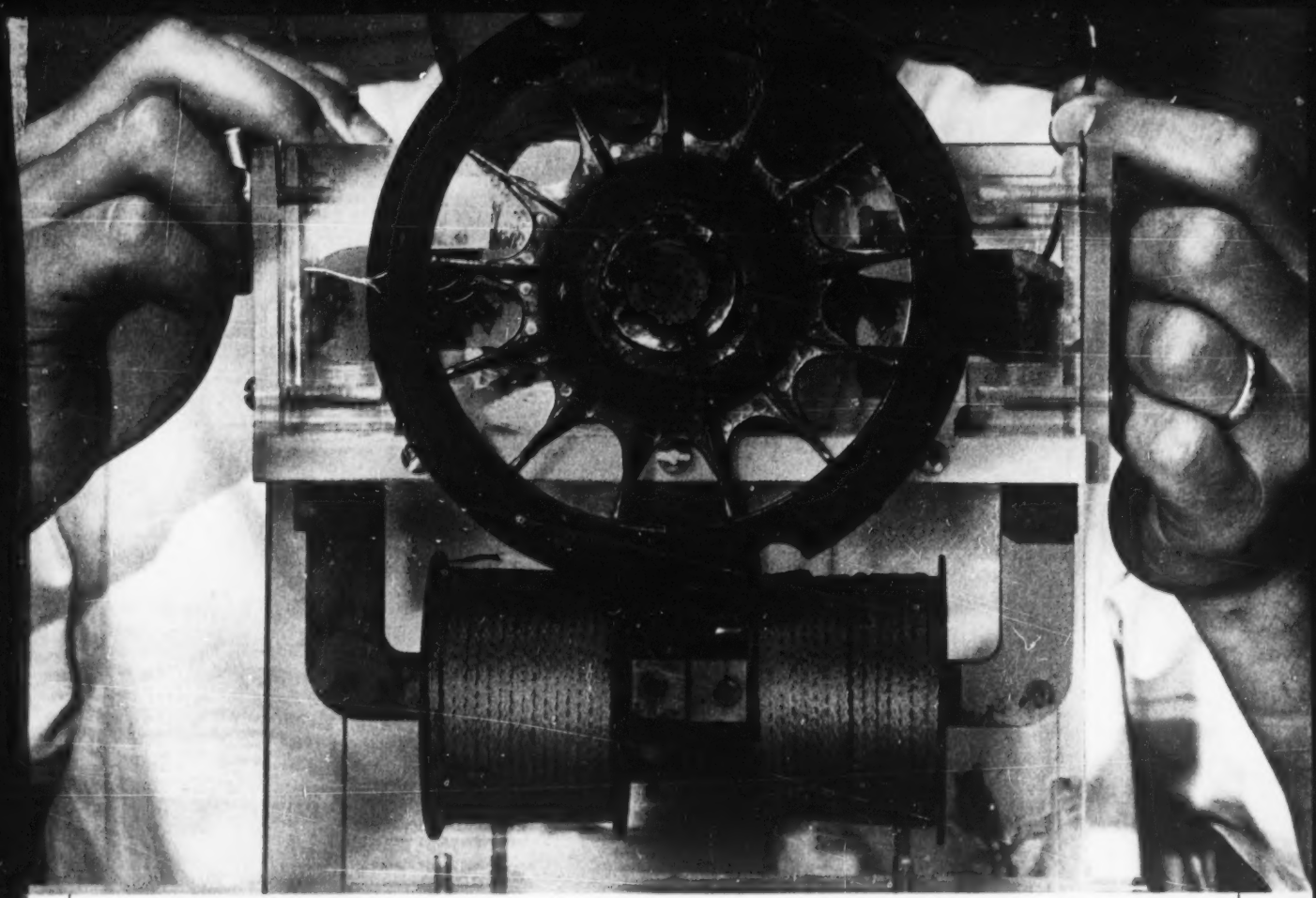
"However, even these obstacles to commercializing superconducting technology are rapidly falling away," says Gregory Yurek, president and CEO of American Superconductor Corp. (ASC), Watertown, MA. The current-carrying limitation of high-T_C materials is caused by a "weak link" between adjacent crystals of the superconducting ceramics. Each crystal conducts electricity without resistance when cooled, but the crystal interfaces don't conduct as efficiently.

Researchers at ASC control the weak-link problem by filling hollow silver tubes with blended bismuth-based superconducting powders and then pulling the tubes through draw dies to fine-wire sizes. The wires are then baked to convert the blended powders into ceramic superconductors. Why this eliminates the weak-link problem is not yet fully understood.

Working with Reliance Electric Co., a Cleveland-based electric motor manufacturer, ASC recently demonstrated a 25-W, 2,500-rpm DC motor containing superconducting coils made from bismuth-based wires. The liquid-nitrogen-emersed high-T_C coils were powered by a 0.5-A current. "This is the first motor using superconducting coils that produces a usable power output," says Yurek.

Development of this motor was funded by the Electric Power Research Institute, Palo Alto, CA. It follows earlier work involving superconducting coils that have been cycled from room temperature to 77 K and continue to perform satisfactory after operating for more than 17 months.

Operating high-T_C materials in liquid nitrogen is convenient and less involved than operating in the liquid helium (4 K) environment required for low-T_C products. However, in high-T_C materials containing bismuth, the



***Technician for Reliance** Electric examines the power leads for a 25-W DC fan motor constructed with two high-T_C superconducting field coils.*

***Conductus's high-T_C** bolometer, packaged in a liquid nitrogen dewar, detects far-IR radiation with greater sensitivity than any other IR detector operating at or above 77 K. Applications include ecological and climatological studies.*

weak-link effect disappears at 20 K.

This also is not yet understood, but researchers are evaluating the use of high-T_C materials at 20 K, where the materials are less likely to lose their superconducting properties in a changing operating environment. Additionally, cryocoolers that refrigerate to 20 K are commercially available for less than $20,000, compared to helium liquefiers that cost more than $200,000. "Being able to flip a switch on a cryocooler is an important step in making superconductor technology commercially feasible," says Alexis Malozemoff, vice president of R&D at ASC.

The flux-creep problem was resolved by introducing small defects in the superconducting bulk material. Defects increase the maximum current a superconductor can sustain, particularly in external magnetic fields. They do so by "pinning," or preventing the motion of magnetic field lines that would otherwise produce electrical resistance.

Current methods for introducing defects into high-T_C materials involve

Processes for developing superconducting integrated circuits have been demonstrated by several companies.

adding impurities which become randomly distributed during processing. Researchers at IBM T.J. Watson Research Center, Yorktown Heights, NY, Oak Ridge (TN) National Laboratory, Argonne (IL) National Laboratory (ANL), Ames (IA) Laboratory, and Iowa State Univ., Ames, have developed a technique that uniformly distributes pinning defects into crystals of Y-Ba-Cu-O.

Defects are introduced into the crystals by bombarding them with a 580-MeV beam of tin ions. The resultant defects are 20 atoms wide and aligned with the direction of the beam. At 77 K, the researchers obtained a critical current density of 450,000 A/cm^2 in a magnetic field of 1 tesla on the modified materials. It should be noted that the crystal interfaces of these Y-Ba-Cu-O materials can sustain only small amounts of current before electrical resistance appears.

High-temperature substrate heater (red disk in picture) was developed to uniformly deposit high-T_C materials, but can be used for other material deposition processes.

Wires made of superconducting materials are becoming commercially available in various current-carrying capacities and in more than 1,000-m lengths. Most of these wires have relatively low critical currents, from 2,000 to 10,000 A/cm^2.

"The heat treatments for these wires get very specific, and companies will not divulge the details so they don't lose their individual competitive advantages," says Roger Poeppel, director of ANL's Superconductivity Pilot Center, one of three such centers established by the U.S. Dept. of Energy to facilitate superconductor technology transfer. A silver-clad bismuth-based superconducting tape for high-field (20 tesla) magnets that is being developed by ANL and Superconducting Products Co., St. Charles, IL, entails extensive heat treating.

"Heat treatments each take about a week, so it takes many months just to do a few trials," says Poeppel. This wire exhibits a critical current of 40,000 A/cm^2 at 4 K. Production doesn't present overwhelming obstacles, once the processing characteristics are defined, Poeppel says.

"Our immediate goal is to make a small coil where the total conductor length is 250 mm," says Poeppel. He estimates that it will take a few more years before this particular product is ready for production.

Argonne researchers also worked with researchers at Westinghouse Electric Corp., Pittsburgh, to develop bulk high-T_C leads that can replace conventional copper leads for transmitting electricity from room-temperature power supplies to low-T_C devices operating at 4 K, such as the coils in MRI systems.

The copper leads used in current low-T_C MRI systems heat up as current is passed through them. The heat is then conducted to the liquid helium refrigerant, causing it to boil off. Collecting and recycling this helium vapor adds considerable cost to conventional systems.

The high-T_C leads developed by Argonne and Westinghouse carry a record 2,000 A without conducting heat back to the liquid helium. They consist of 20 rectangular bars fabricated from a composite of silver and Y-Ba-Cu-O.

One of the first commercially available high-T_C products also used Y-Ba-Cu-O materials as a principle component in the fabrication of a cryogenic liquid level sensor by researchers at Argonne and Illinois Superconductor, Evanston, IL. The sensor consists of two wires with a measurable resistance. When the wires are immersed in a cryogenic liquid, the exposed portion retains its resistance and the immersed portion loses its resistance. The overall resistance then can be calibrated to the liquid level.

This product has extensive applications in medical life sciences, biotechnology, and cryopreservation industries. A similar stand-alone system has recently been announced by Lake Shore Cryotronics, Westerville, OH.

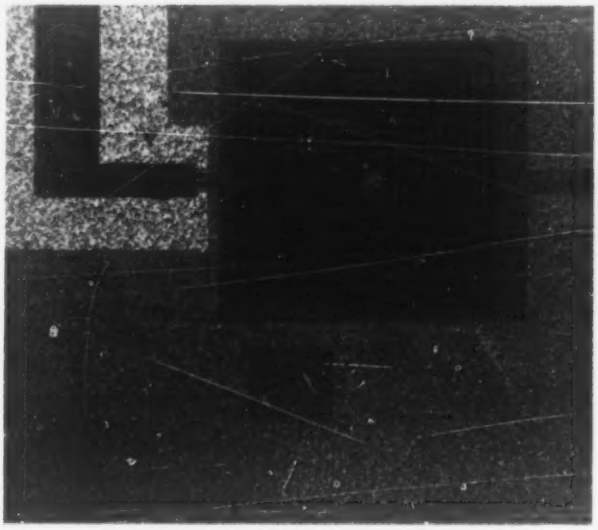
While there is much new in the bulk high-T_C arena, steady progress has also been made in thin films.

Three companies—Biomagnetic Technologies Inc., San Diego, Bellcore, Red Bank, NJ, and Conductus, Sunnyvale, CA—all recently announced processes for manufacturing Josephson junctions from thin-film Y-Ba-Cu-O superconductors. All the processes are repeatable, inexpensive, and use conventional semiconductor fabricating techniques.

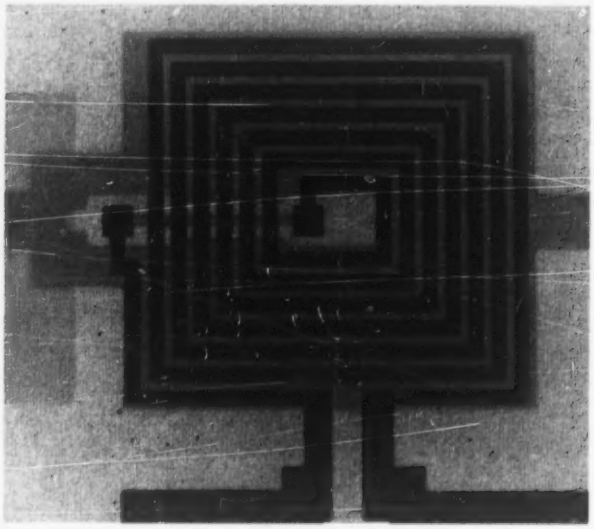
Josephson junctions are the superconducting equivalent of the transistor, which is the basis for any future superconducting circuits. They are formed from two weakly coupled superconductors that switch from the superconducting state to the nonsuperconducting state in about a trillionth of a second.

Researchers at Conductus fabricated the first integrated circuit, a SQUID magnetometer, containing devices made from high-T_C superconductors. SQUIDs (superconducting quantum interference devices) are the most sensitive magnetic field devices known. They operate by converting hard-to-measure magnetic flux into

High-T_C infrared detectors are up to 50 times more sensitive than current room-temperature devices.



***Integrated SQUID** (superconducting quantum interference device) and flux transformer made with a seven-layer epitaxial process containing three high-T_C layers.*



***The process used** in making this five-layer high-T_C integrated SQUID magnetometer and flux transformer has been used to deposit up to 15 insulating and superconducting oxides.*

easy-to-measure voltage. Conductus's SQUID sensor consists of a superconducting loop containing two Josephson junctions.

The high sensitivity of Conductus's SQUID is due to a superconducting flux transformer that has five- or 10-turn coils, coupled to a single-turn 10-mm-dia sensing loop.

"This circuit contains seven layers of material, making it the most complex circuit ever fabricated from high-T_C materials," says John Rowell, president and CEO of Conductus. "We can currently deposit 15 layers of epitaxial oxides, both high-T_C superconducting and insulating, plus a silver contact layer. These circuits demonstrate all the elements and techniques required to build integrated circuits for signal processing and digital applications."

Conductus's fabrication process uses conventional semiconductor lithography to pattern each layer and laser ablation to deposit the Y-Ba-Cu-O films on sapphire substrates.

Josephson junction devices also have been fabricated from bismuth-based high-T_C materials using low-temperature chemical vapor deposition methods by researchers at Fujitsu, Kawasaki, according to a report in the Nikkan Kogyo Shimbun, a Japanese newspaper.

Thin films of high-T_C Y-Ba-Cu-O also are being used as far-IR detectors (wavelengths >20 μm) in bolometer devices packaged in a liquid nitrogen dewar.

A bolometer is a device that measures small amounts of radiation by determining the changes of resistance in a conductor caused by changes in its temperature. The detector has up to 50 times the sensitivity of current room-temperature detectors. Applications include chemical laboratory spectroscopy, strategic defense, space-based environmental, ecological, and climatological studies.

There are a large number of additional thin-film applications for which products are not yet available, but for which prototypes have been demonstrated. Passive microwave (MW) devices are one application area that offers a near-term market for high-T_C electronics. The low-loss characteristics of superconductors enable MW engineers to design ultra-high-frequency devices with improved sensitivity, higher resolution, and with lower power consumption.

High-T_C interconnects for integrated circuits is another strong application area. Both Hitachi, Tokyo, and IBM's Research Division, Yorktown Heights, NY, are working on projects to operate their CMOS (complementary metal-oxide semiconductor) DRAM circuits at 77 K. Operating in this environment cuts the gate delays in half and therefore can nearly double the speed of a computer.

An offshoot of thin-film research is the development of equipment to apply the films. For the most part, high-T_C thin-film materials have been fabricated with existing semiconductor-based equipment. However, specialty equipment that had to be developed is now available for use in other areas.

In order to apply uniform films of high-T_C materials, for example, a heater was developed that allows precise and stable temperature control in environments from UHV to atmospheric pressures. The 7.5-cm heater from U.S. Inc., Campbell, CA, operates up to 950 C and holds the temperature stable to +/−2 C.

The current component design and fabrication stage of the Superconducting Supercollider project and the increasing popularity of MRI systems are the biggest technology drivers for high-T_C materials.

Continued improvements in wire performance and fabrication techniques and in thin-film processing should further the growth in high-T_C materials and further products throughout this decade. **R&D**

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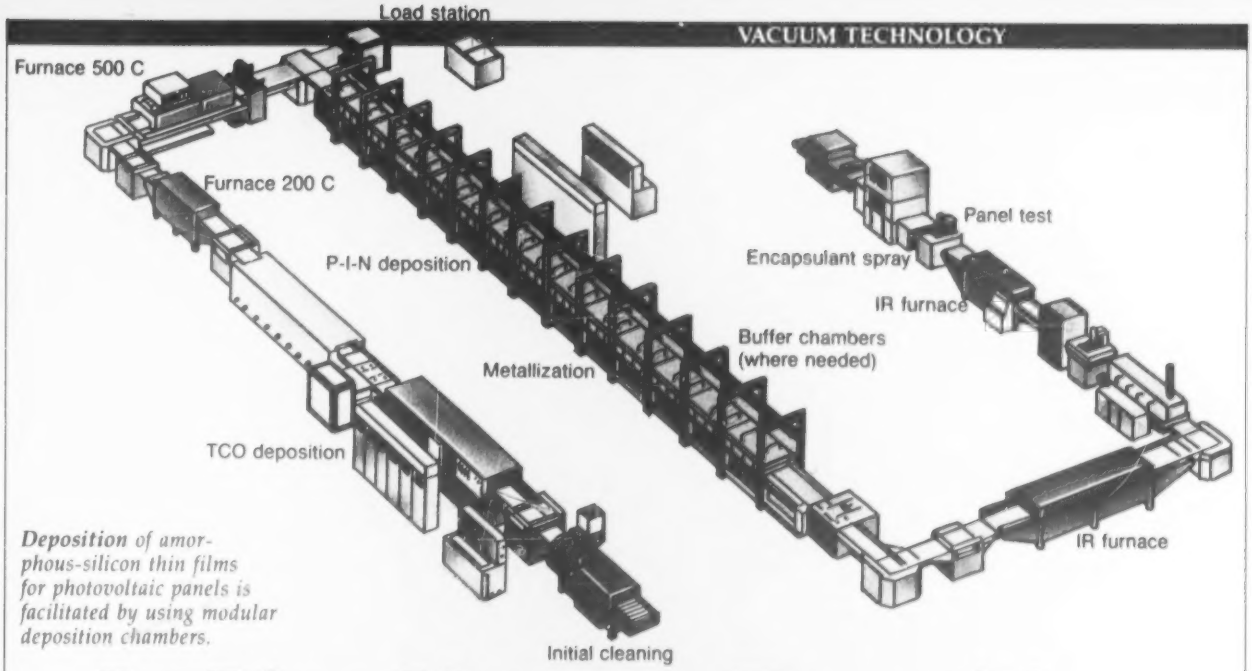
High **471** Medium **472** Low **473**



Deposition of amorphous-silicon thin films for photovoltaic panels is facilitated by using modular deposition chambers.

In-Line Vacuum Deposition Needs Special Solutions

Depositing large thin-film areas of amorphous silicon requires development of a continuous process for high throughput.

*By **J. Grant Armstrong**, MKS Instruments, **Pawan Bhat**, Materials Research Group Inc., and **Scott Brown**, Glasstech Solar Inc.*

R&D

THE AUTHORS

J. Grant Armstrong is a regional manager at MKS Instruments, Boulder, CO. **Pawan Bhat** is president of Materials Research Group Inc., Wheatridge, CO, and previously was director of technology at Glasstech Solar Inc., Golden, CO. **Scott Brown** is president of Glasstech Solar Inc.

Uniform deposition of large-areas of amorphous-silicon thin films, such as those used in flat-panel displays and photovoltaic (PV) panels, is difficult enough in a laboratory batch operation. But the process and yield demands needed to scale up to production levels add yet another level of problems.

These problems include development of a vacuum system for pumping across a series of chambers, continuous operation of various deposition processes, and minimization of defects in the films.

The most common method to deposit amorphous-silicon thin films is plasma-enhanced chemical vapor deposition (PECVD). Since a major factor in production improvements comes from the process equipment, systems must be designed for continuous processing, automatic operation, and minimum downtime.

Ideally, raw material should go in one end and a finished product should come out the other. This concept leads to a continuous in-line configuration consisting of a series of PECVD chambers with load locks at each end.

Severe demands are placed on the vacuum pumping systems for these systems. Each chamber must achieve good base pressures, handle high gas loads, provide rapid pumpdowns, and accommodate frequent cycling.

Our systems use mechanical pumps and blowers for high-pressure processes and turbopumps for high-vacuum applications. Holding those vacuums while running the PECVD processes continuously with few defects is the key to obtaining high net throughputs.

Just one defect in the amorphous-Si thin film of a flat panel display can render an entire panel unusable. One inoperative transistor can create a line across the panel or a visible dark spot on an active matrix panel.

Similarly, the overall performance of amorphous-Si PV panels, which can be as large as 1.2 × 2.4 m, can be severely degraded by a 5-μm defect, since the lowest performance cell in a series of cells dictates the performance of the entire panel.

Fast-acting rectangular gate valves isolate each of our in-line chambers and open only long enough, typically about 5 sec, to allow the 1- to 5-mm-thick glass substrates to move through.

Buffer chambers between some

A few parts per million of one dopant in the wrong layer of an amorphous-silicon thin film could make a PV cell unusable.

deposition processes limit cross-contamination of dopant gases to adjacent chambers. Conventional in-line systems do not use buffer chambers—the processes in each chamber stop when substrates are transported through the system.

Fabricating a PV panel requires three consecutive PECVD steps with buffer chambers. The first step is a 100-Å-thick deposition of p-doped amorphous-Si. The second is a 3,000-Å intrinsic amorphous-Si layer, and the third is a 200-Å n-doped amorphous-Si layer.

A few parts per million of a dopant, such as phosphorous or boron, in the wrong layer of these films could render the entire PV cell unusable.

As the substrate nears the end of the first deposition process, a gate valve opens, and the substrate moves into a buffer chamber. Just before this valve opens, the buffer chamber pressure is equalized to that of the first deposition chamber. As the substrate moves through the buffer chamber, contaminating gases are removed and the pressure is equalized to that of the next process chamber.

To maintain a continuous process, pressure measurement and control are critical elements in this system. We use temperature-regulated Baratron capacitance manometers to measure pressures from 0.1 to 1 torr.

To ensure that process gases do not diffuse from one chamber into another when a gate valve opens, the adjoining chambers must be at the same pressure. Even a pressure difference of 10 mtorr could cause process gases to poison a buffer chamber, or cause the buffer gases to interfere with an active plasma in an adjoining chamber.

Since the gate valves are open only about 5 sec, the pressure control system must react quickly to achieve the needed pressure. Exhaust throttle valves are used to control the chamber pressures, since these devices provide a stable control and respond to pressure changes in a few seconds.

This valving system also ensures that the process gases have enough residence time in each chamber to react with each other and the substrate, by allowing gas flows to be set upstream and pressures to be set with the downstream throttle valve.

To minimize contamination from background gases, low base pressures of H_2O, O_2, N_2, and C are required. This dictates using specific materials to fabricate the equipment. Stainless steel is used wherever possible, although Al and Cu can be used in some situations. Metal-sealed CF vacuum flanges are used to minimize leaks or permeation of gases through O-rings and metal-sealed VCR fittings are used in gas lines.

Thermal mass-flow meters and controllers are used to control up to eight gases flowing into each chamber. A proprietary gas distribution system uniformly distributes gases over the substrates.

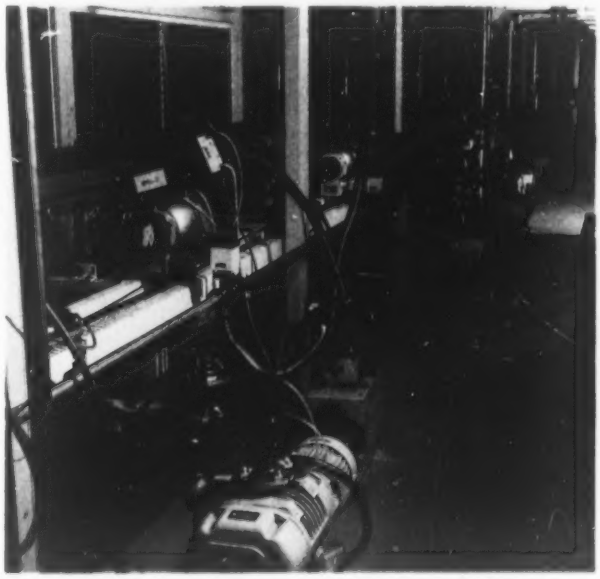
The chambers, pumping systems, and radio-frequency-powered plasma electrodes were designed to confine plasmas to localized deposition areas. This confinement minimizes deposition outside the desired area.

Interaction of the plasma with the PECVD chamber walls can cause substrate contamination where doping gases are used, but this can be avoided by changing gas sources.

High-vacuum gages monitor base pressures and must tolerate many cycles from low to high pressures in an in-line system. Process chemistries that use corrosive gases are especially hard on these gages.

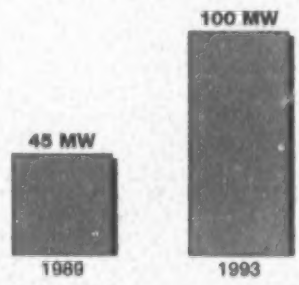
Hot-filament gages have been used with some success. However, future systems may employ cold-cathode gages to improve gage lifetimes. Partial-pressure instruments are placed at key points in the system to monitor the processes and provide diagnostics.

For the most part, large-area process systems can use readily available standard components. Equipment manufacturers have built, or plan to build, cluster tools or continuous in-line systems. Additional design considerations include design of transport systems that withstand more than 300 C and materials for electrodes. Many of these issues have been addressed by manufacturers, but more work needs to be done. R&D



***Turbomolecular pumps** (spherical housings) provide 10^{-7} torr for plasma-enhanced chemical vapor deposition in this in-line continuous deposition system for fabricating PV panels.*

Sunny Days Ahead for Photovoltaic Panels



Source: PV News

***More than** 22% annual growth forecast in worldwide shipments of PV modules is driving development of new applications for amorphous silicon thin films.*

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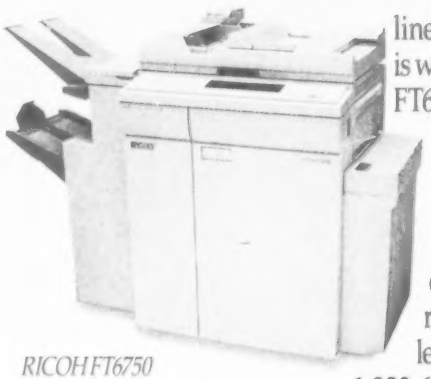

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